

FIG. 1

MORALES, KLR, ETH-SIOH-1 IN CDC13

EXPL S3PUL

SAMPLE		DEC. & VT	
DATE	FEB 27 98	DFRQ	499.699
SOLVENT	CDC 13	DN	HL
FILE	EXP	DPWR	20
ACQUISITION		DOF	6
STFRQ	499.699	DM	NNN
TN	111	DMM	C
AT	3.277	DMF	200
NP	39296	DSEQ	
SW	5996.1	DRES	1.0
FB	3400	HOMO	N
BS	16	DEC2	
TPWR	63	DFRQ2	0
PW	4.7	DN2	
DL	0	DPWR2	1
TOF	0	DOF2	0
NL	400	DM2	N
CT	160	DMM2	C
ALOCK	N	DMF2	200
GAIN	NOT USED	DSEQ2	
FLAGS		DRES2	1.0
11	N	HOMO2	N
LN	N	PROCESSING	
DP	Y	16	6.30
HS	NN	WTFILE	
DISPLAY		PROC	FT
SP	-138.2	FN	NOT USED
WP	5133.1	MATH	R
V\$	8848		
SC	0	WERR	
WC	250	WEXP	
NIMM	20.53	WBS	
LS	33.57	WNT	
RFL	4131.0		
RFP	3627.8		
TH	7		
INS	1.000		
NM	PH		

FIG. 1A

¹H NMR SPECTRUM (500 MHz) OF ETM-SiOH-1 (NON-POLAR IMPURITY) IN CDCl₃.

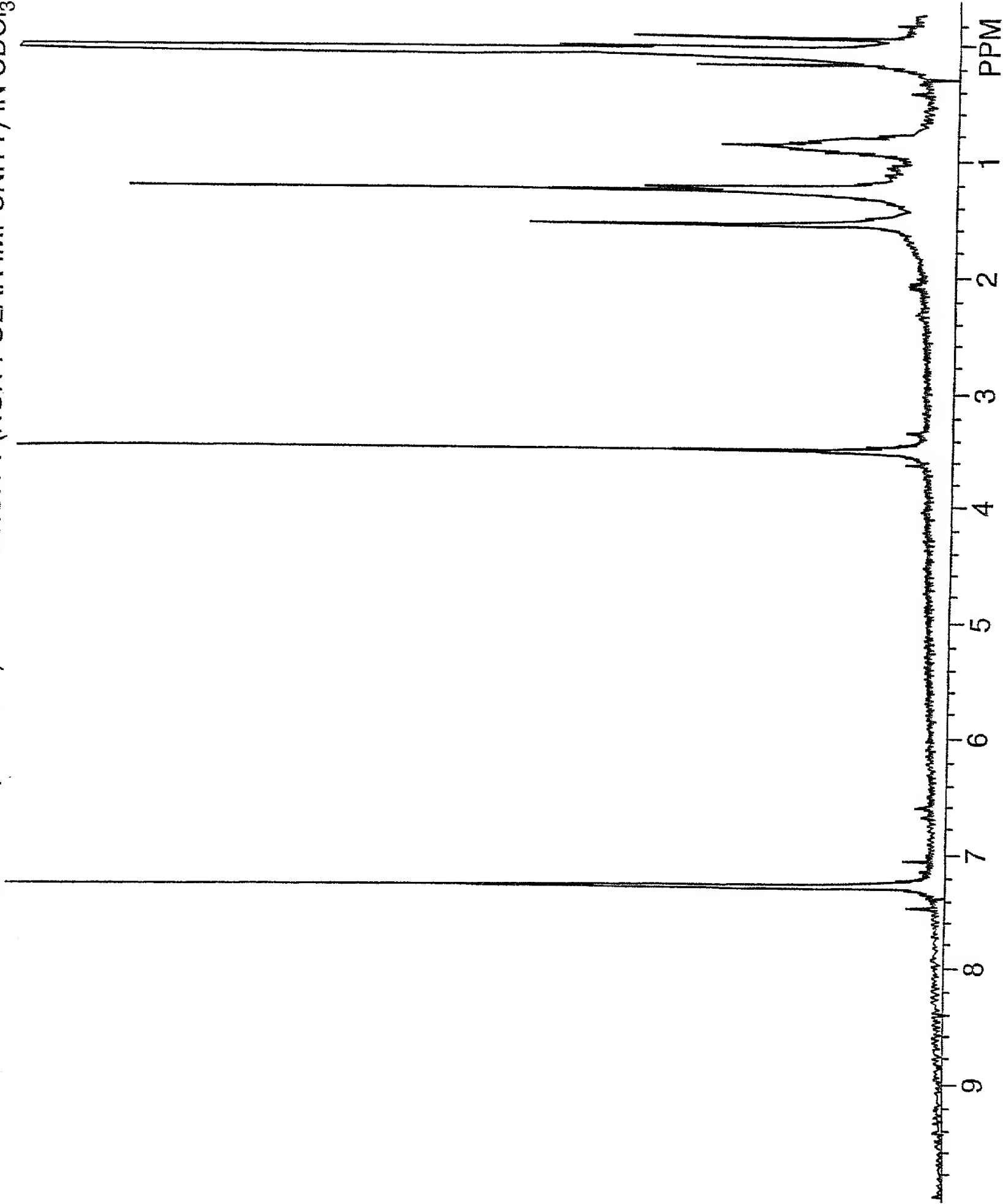


FIG. 1B

0 20 40

ETM-SiOH-5-HP1
(ETM 775)

ETM-SiOH-5-HP2

.02

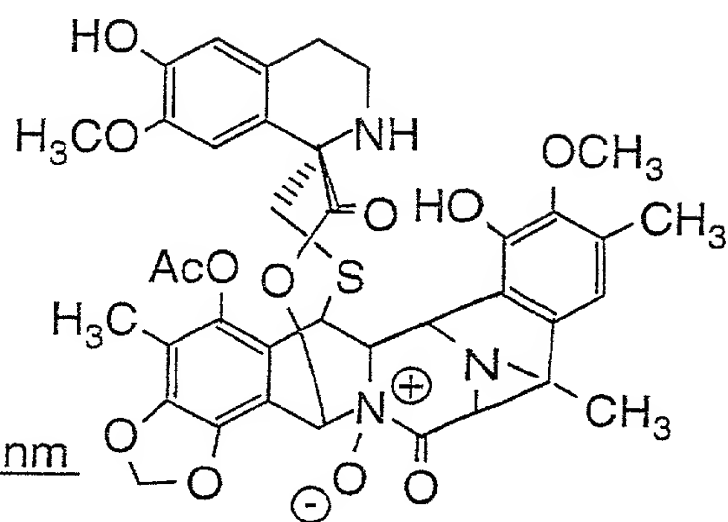
.5
ALL

0 20 40

TIME 0 --- 40 MIN

HPLC CONDITIONS

DETECTOR: DAD

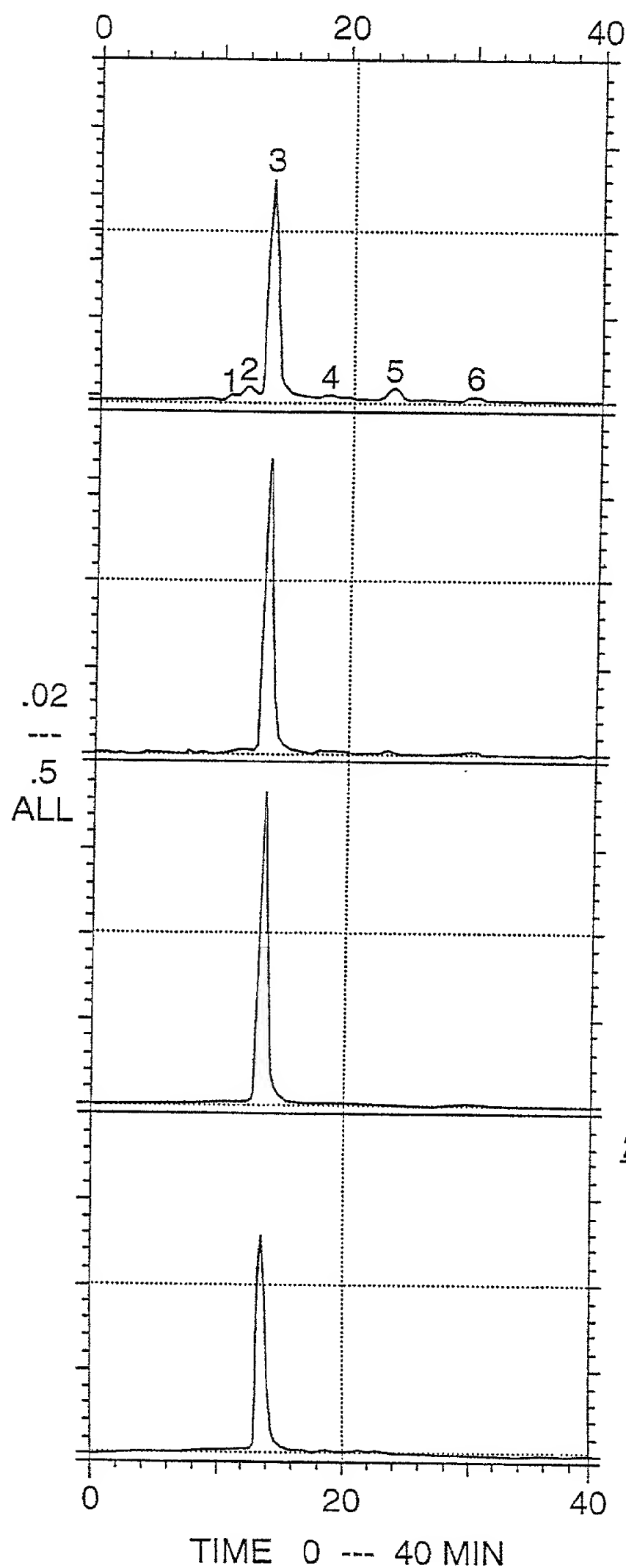


ETM 775

FIG. 2

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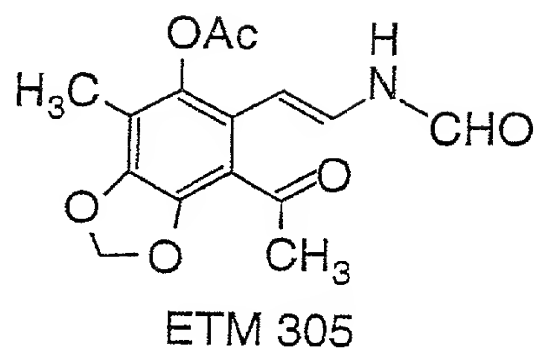
HPLC CHROMATOGRAM OF ETM-SiOH-3 (ETM 305)



210 nm

FIG. 3

220 nm



254 nm

280 nm

HPLC CONDITIONS

COLUMN: Phenomenex/Ultracarb
5 ODS. ID 150 x 10 mm

MOBILE
PHASE: 3:1 MeOH/H₂O 0.02 M NaCl

FLOW RATE: 1mL/min

DETECTOR: DAD

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HPLC CHROMATOGRAM OF ETM-SiOH-2 (TRACE METABOLITES).

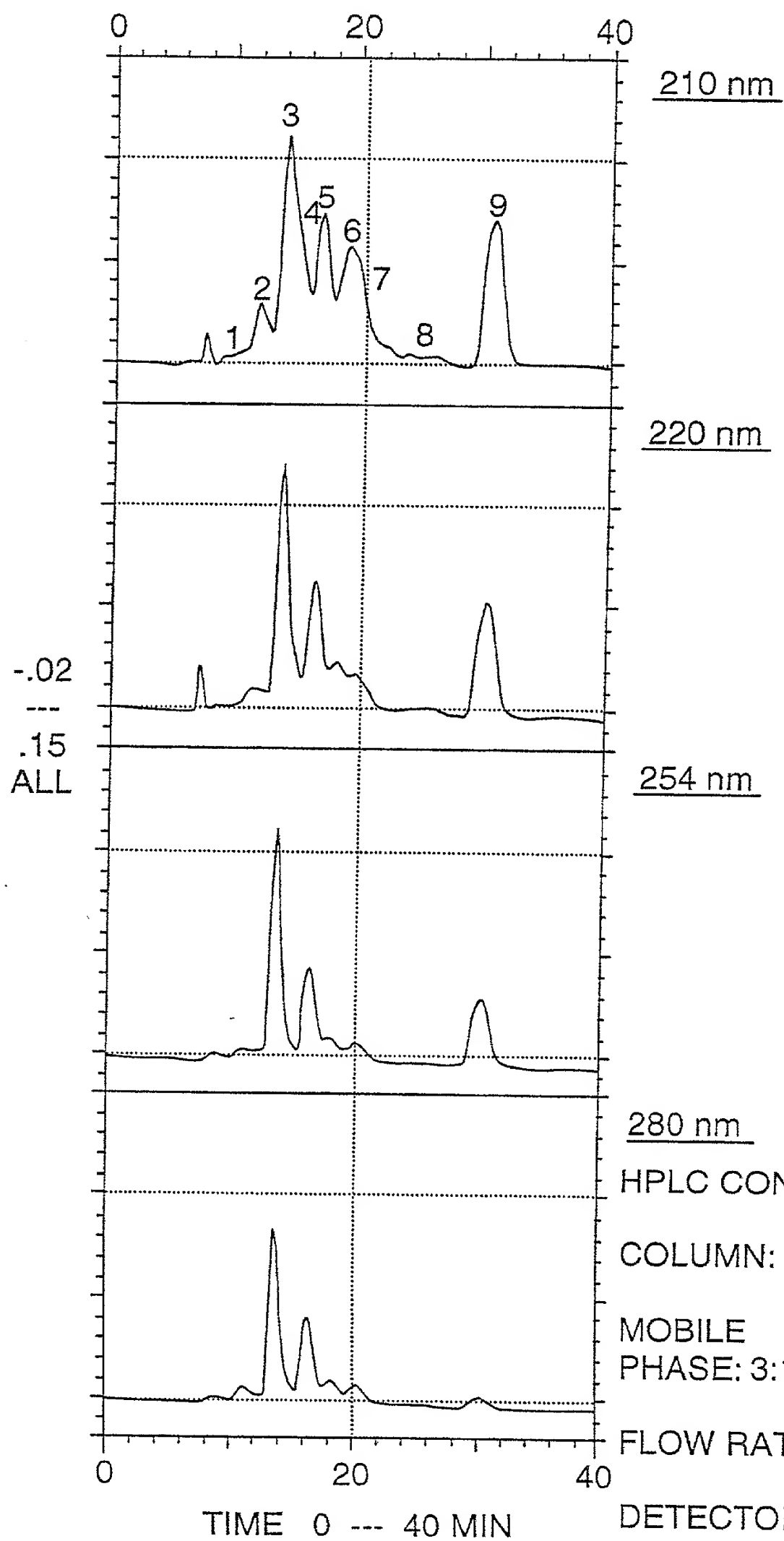


FIG. 4

LRFAB MASS SPECTRUM OF ETM 305 IN M.B. (MAGIC BULLET⁴)

FILE: ETMSIOH4HP1 IDENT:2 ACQ: 16-DEC-1997 12:43:40 +0:45 CAL:CSI121697
 ZAB-SE FAB+ MAGNET BpV: 15.3V TIC:2105487744 FLAGS:HALL
 FILE TEXT: MORALES ETM-SIOH-4-HP1 IN M.B.

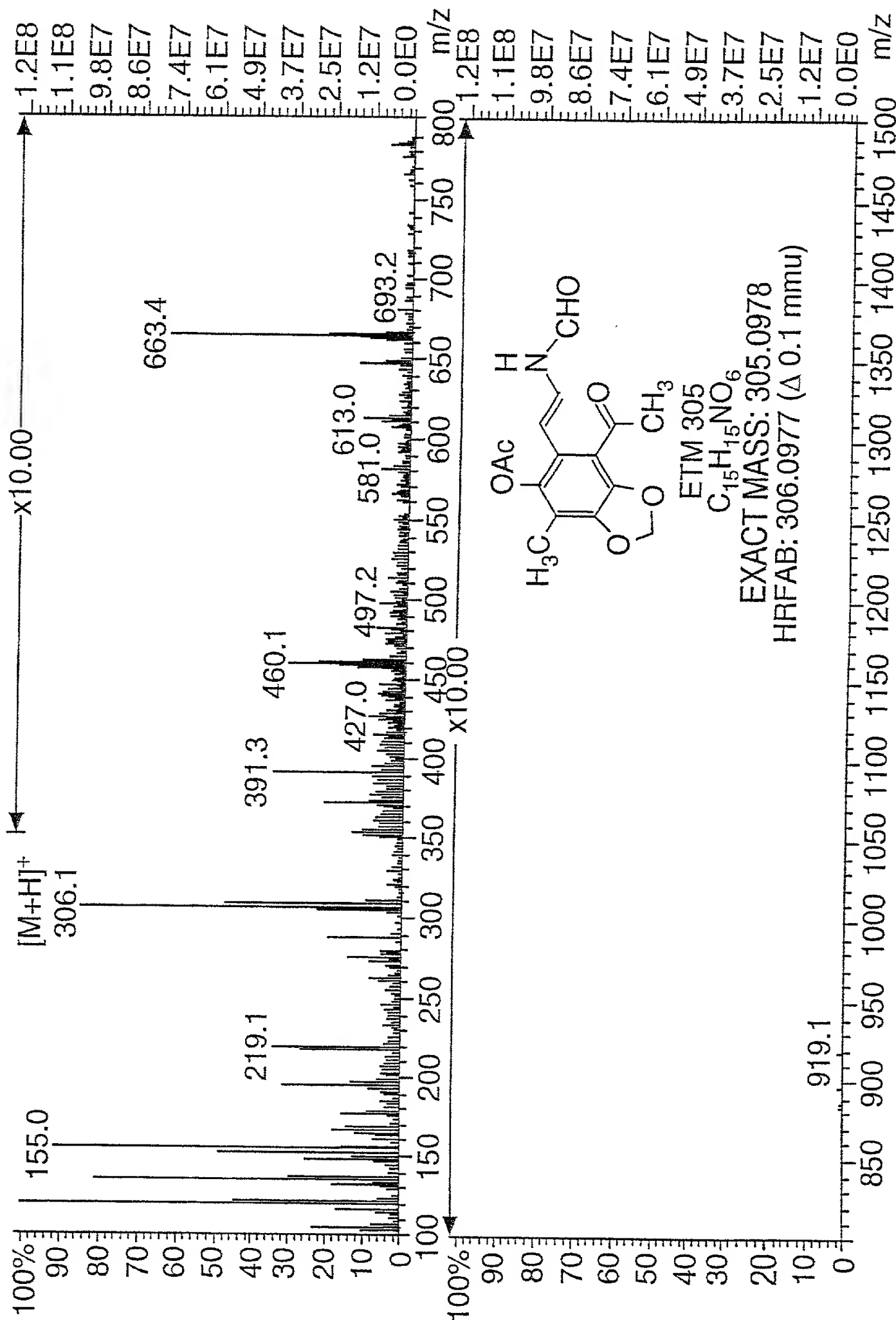


FIG. 5

ESI MASS SPECTRUM OF ETM 305.

etm-sioh-4hp1
Q5689 7 (1.192) Sm(Mn, 2x0.40): Cm (5:9-1:4)

Scan ES+
1.91e6

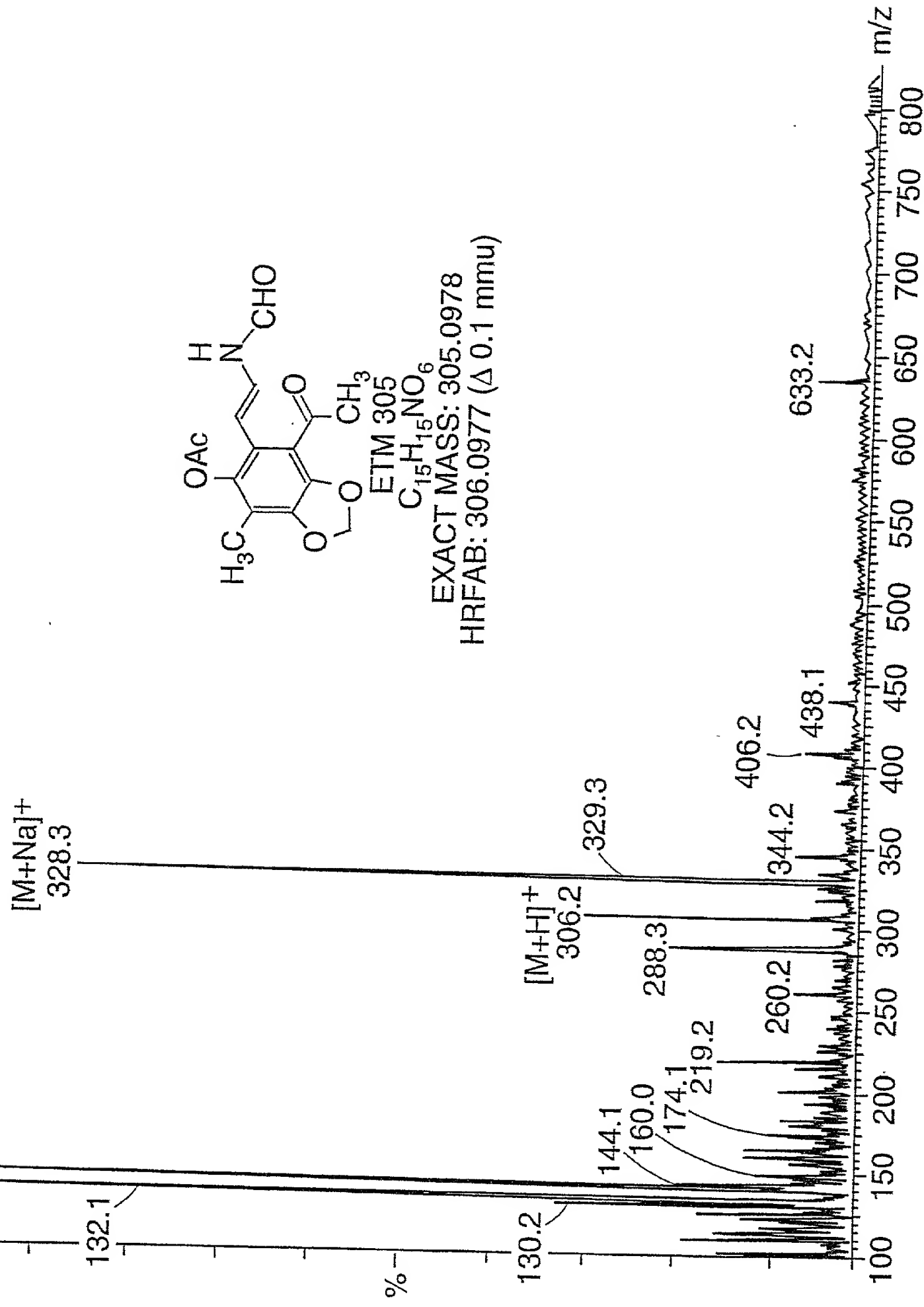


FIG. 6

1-UV in CD3OD F2:750.107 s2puf

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H NMR SPECTRUM (750 Mhz)
OF ETM 305 IN CD₃OD.

CC(=O)Oc1cc(C)c(C(=O)Nc2cc(C)c(C(=O)OC)c2)c1

ETN 305

*NUMBER IN PARENTHESIS
CORRESPONDS TO δ IN Et 743.

Chemical structure of ETN 305 is shown, with protons labeled H_a and H_b. The structure includes a benzene ring with a methoxy group, a methyl group, and a side chain containing a methyl ketone and an amide group.

Key peaks in the spectrum are labeled:

- NHCHO δ 8.04
- H_a δ 7.09, $J = 15$ Hz
- H_b δ 6.19, $J = 15$ Hz
- O-CH₂-O- δ 6.09 (6.11, 6.01)
- ArCOCH₃ δ 2.28 (2.29)
- ArCOCH₃ δ 2.52
- Ar-CH₃ δ 2.04 (2.03)

1-UV in CD3OD F2:750.107 s2puf

SW1: 7499	QEL: 3770.1	USER: --DATE: DEC 27 1997
PW: 9.0 usec	NA: 1768	PTSId: 32768
PD: 0.0 sec	LB: 0.0	WinNuis-\$ETM-UVI.fid

FAB/MS/MS SPECTRUM OF ETM 305.

70-4SE FAB+ E2B2 BpM:306 BpV:31.1V TIC:124301384 FM:306.10 FLAGS:NORM
FILE TEXT: MORALES ETM-UV MSMS ON 306.1 CELL 0.5 HE 90% MORALES ETM-UV MSMS ON 306.1 CELL 0.5 HE>

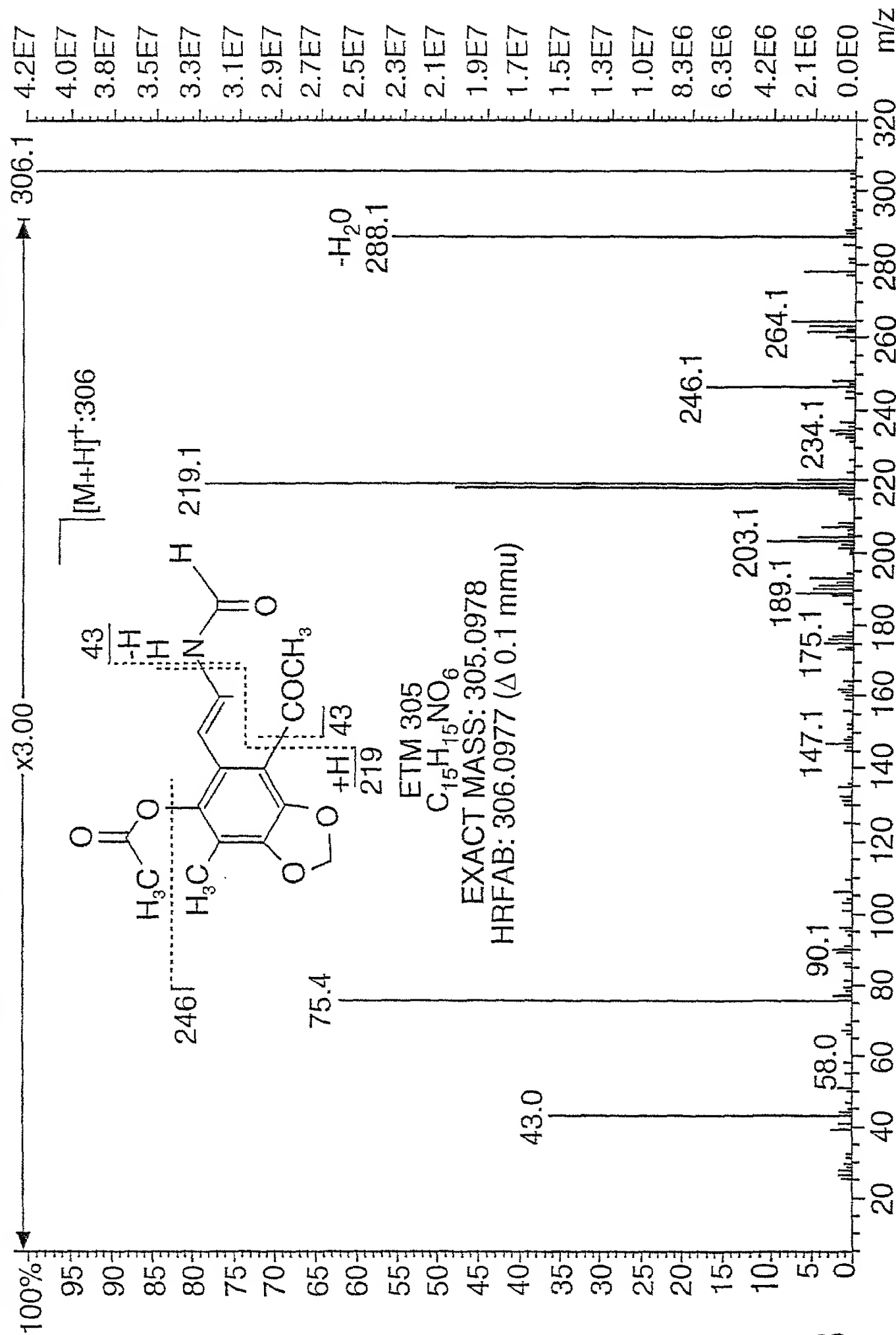
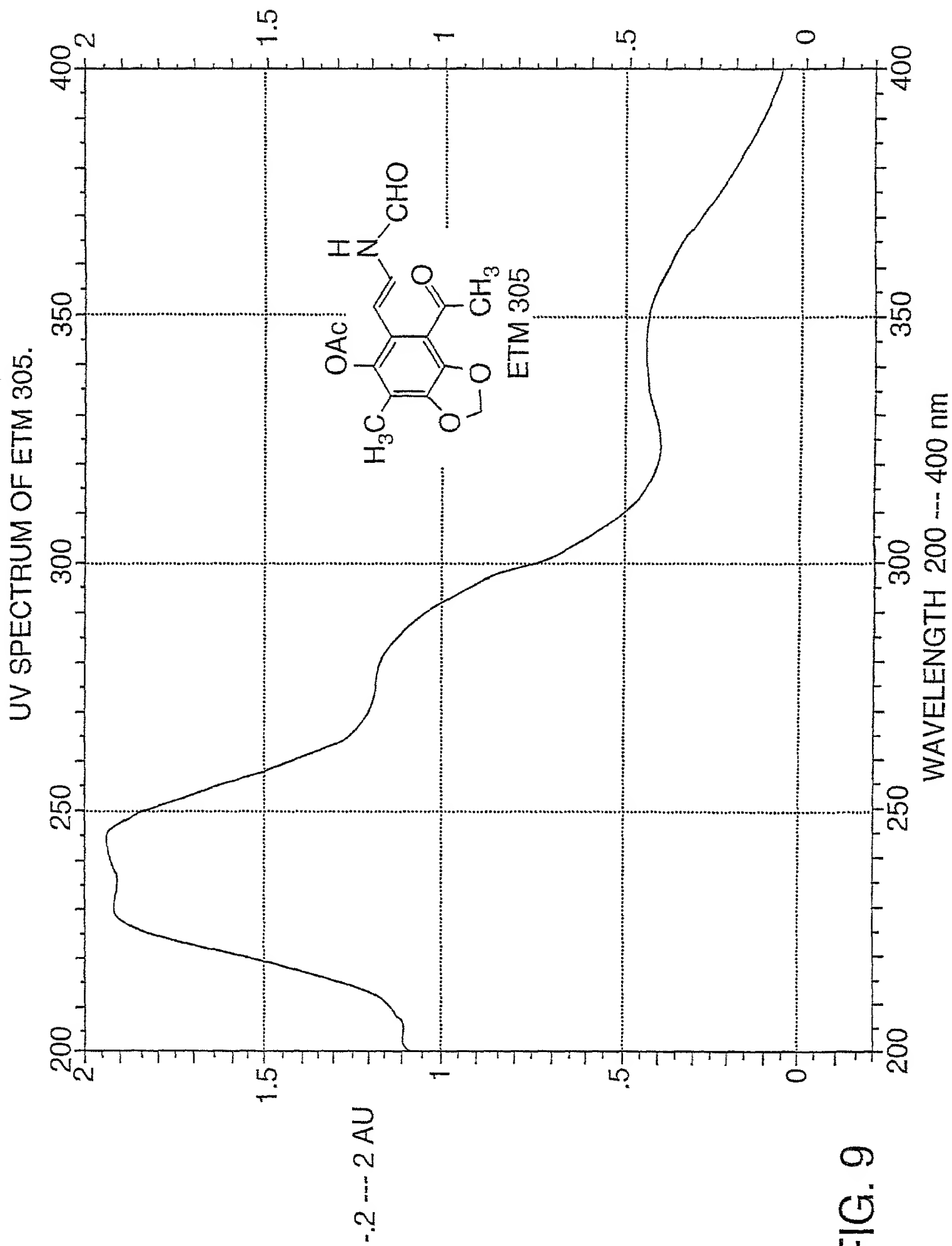


FIG. 8

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UV SPECTRUM OF ETM (PHARMAMAR).

INT OF WINDOW 39: UV APEX SPECTRUM OF PEAK 7.82 OF PICO-M2.D

UV. APEX SPECTRUM OF PEAK 7.82 OF PICO-M2.D

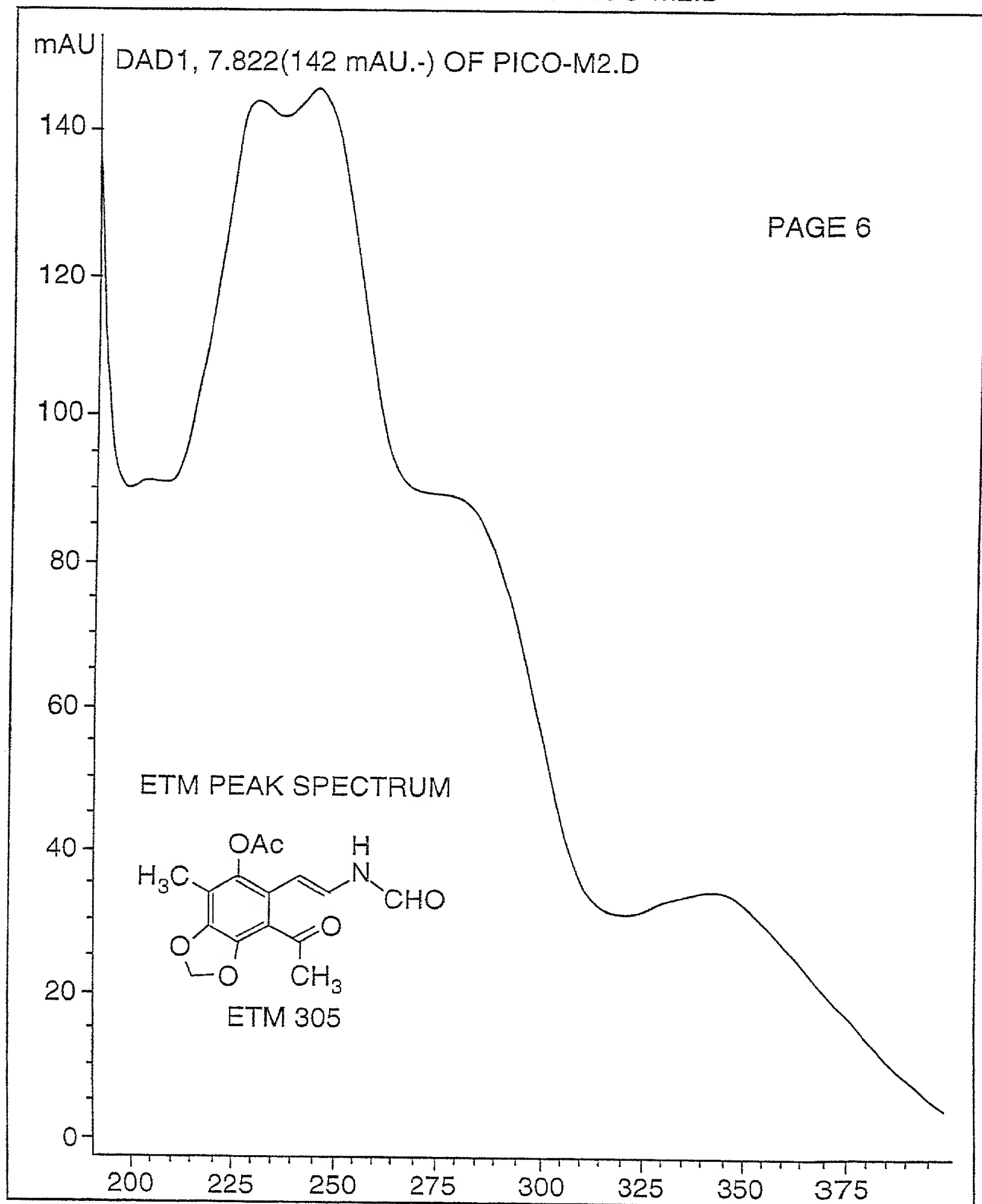


FIG. 10

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LRFAB MASS SPECTRUM OF ETM 775 IN M.B.

FILE: ETMSIOH5HP1 IDENT:2 ACQ: 16-DEC-1997 12:46:41 +1:00 CAL:CSI121697
ZAB-SE FAB+ MAGNET BpV: 15.3V TIC:1626444160 FLAGS:HALL
FILE TEXT: MORALES ETM-SIOH-5-HP1 IN M.B.

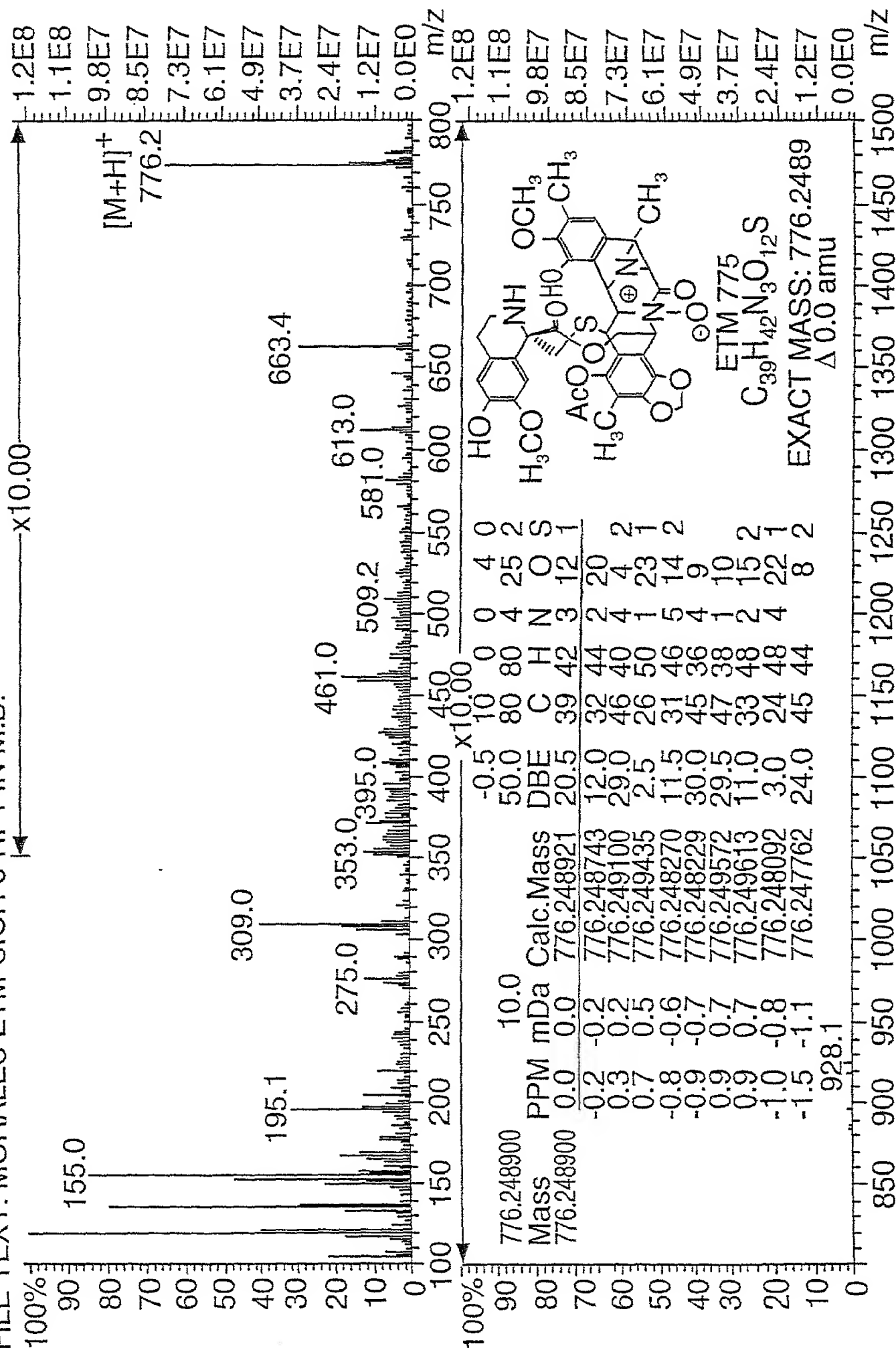


FIG. 11



Scan ES-
1.07e5

ESI MASS SPECTRUM OF ETM 775 (NEGATIVE MODE).

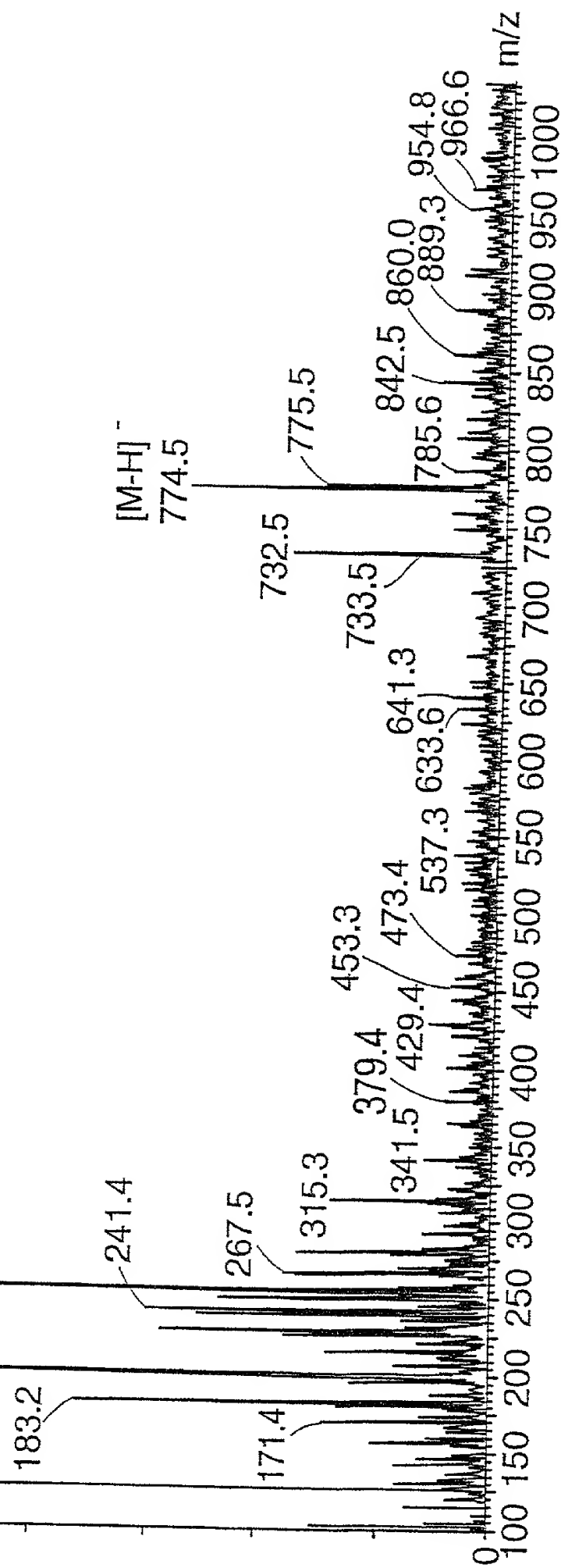
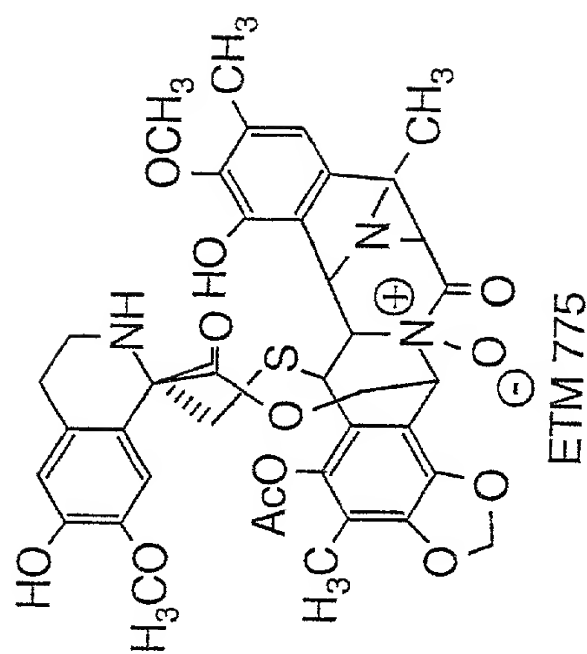


FIG. 13

FAB/MS/MS SPECTRUM OF ETM 775 (m/z 138-302).

FILE: MS9082 IDENT:1 SMO(1,5) PKD (5,3,5,0.00%,0.0,0.00%,F,F) SPEC (HEIGHTS, CENTROID) ACQ:3-DEC-97>
70-4SE FAB+ E2B2 BpM:777 BpV:146.3V TIC:371164064 FN:776.25 FLAGS:NORM

FILE TEXT: MORALES SFIM2275 MSMS ON 776.25 CELL 0.5 HE 90% MORALES SFIM2275 MSMS ON 776.25 CELL>

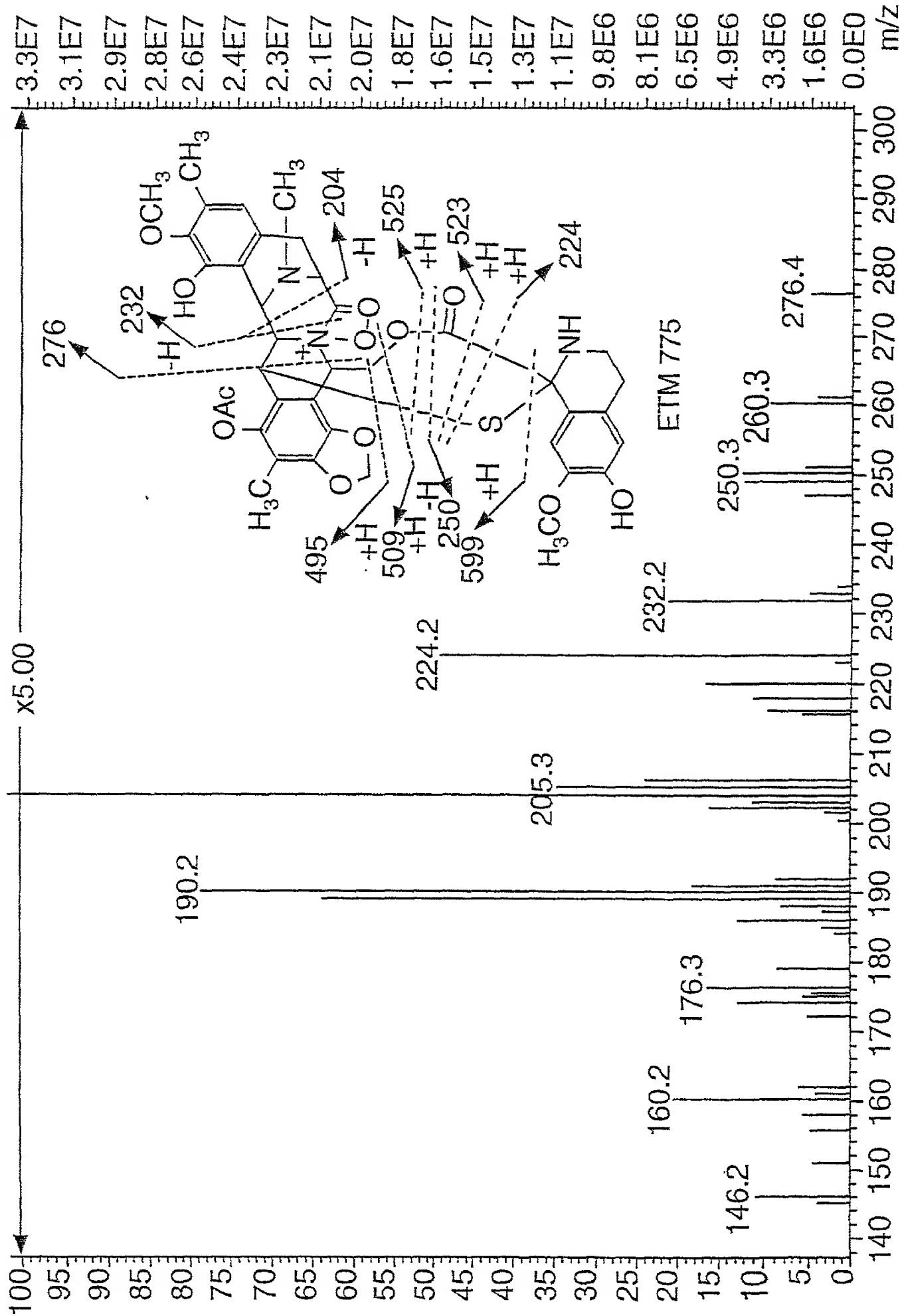


FIG. 14

FAB/MS/MS SPECTRUM OF ETM 775 (m/z 440-620).
 FILE: MS9082 IDENT:1 SMO(1,5) PKD (5,3,5,0.00%,0.0,0.00%,F,F) SPEC (HEIGHTS, CENTROID) ACQ:3-DEC-97>
 70-4SE FAB+ E2B2 BpM:777 BpV:146.3V TIC:371164064 FN:776.25 FLAGS:NORM
 FILE TEXT: MORALES SFIM2275 MSMS ON 776.25 CELL 0.5 HE 90% MORALES SFIM2275 MSMS ON 776.25 CELL>

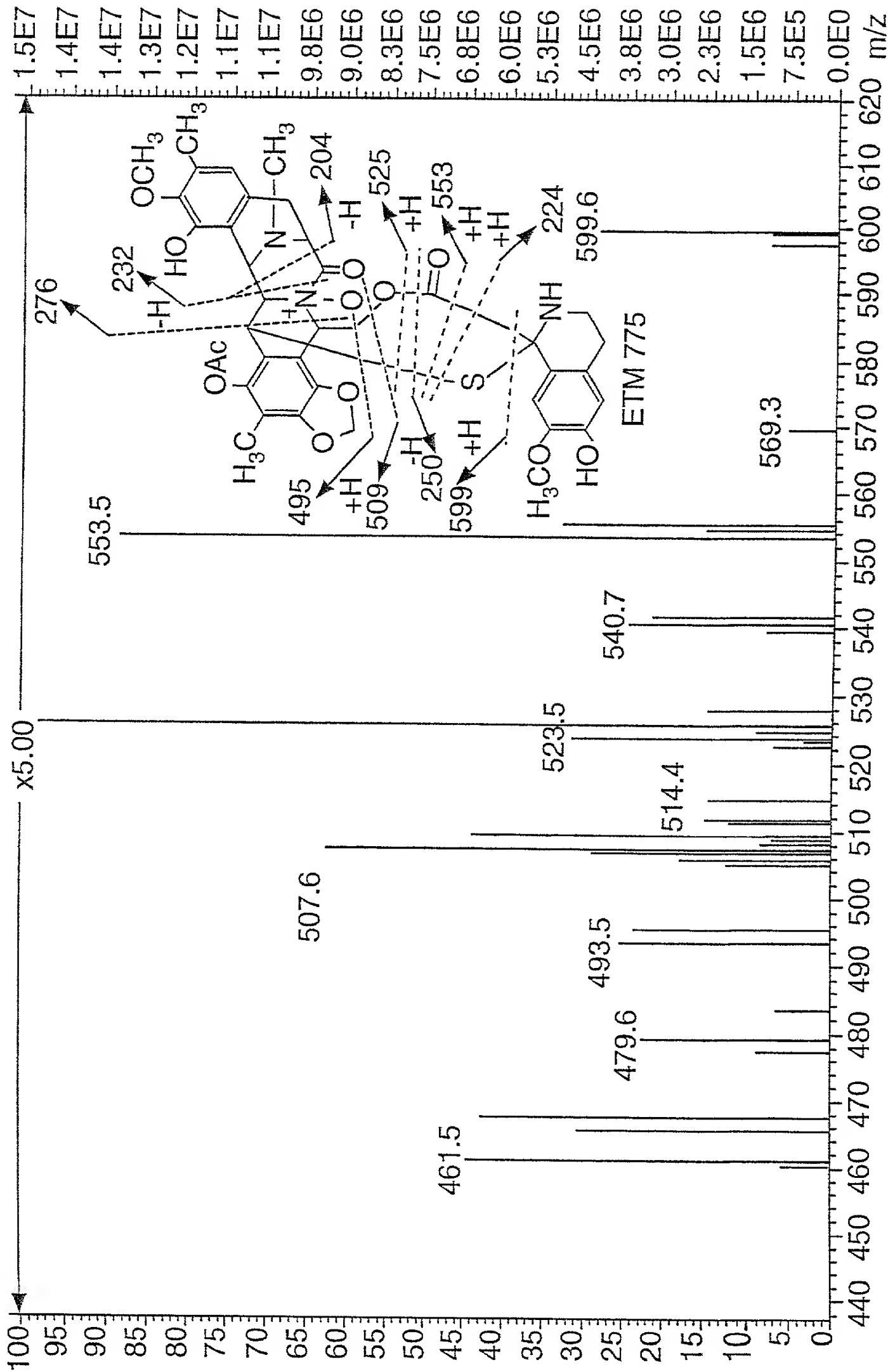


FIG. 15

H NMR SPECTRUM (750 MHz) OF ETM 775 IN CD₃OD.

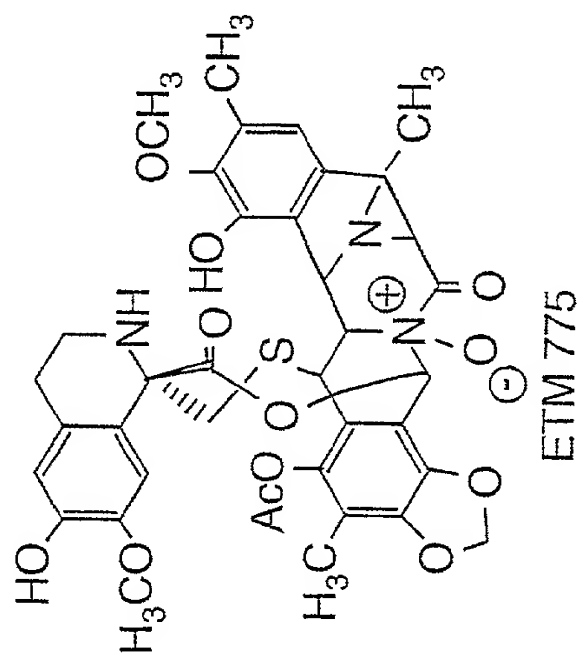
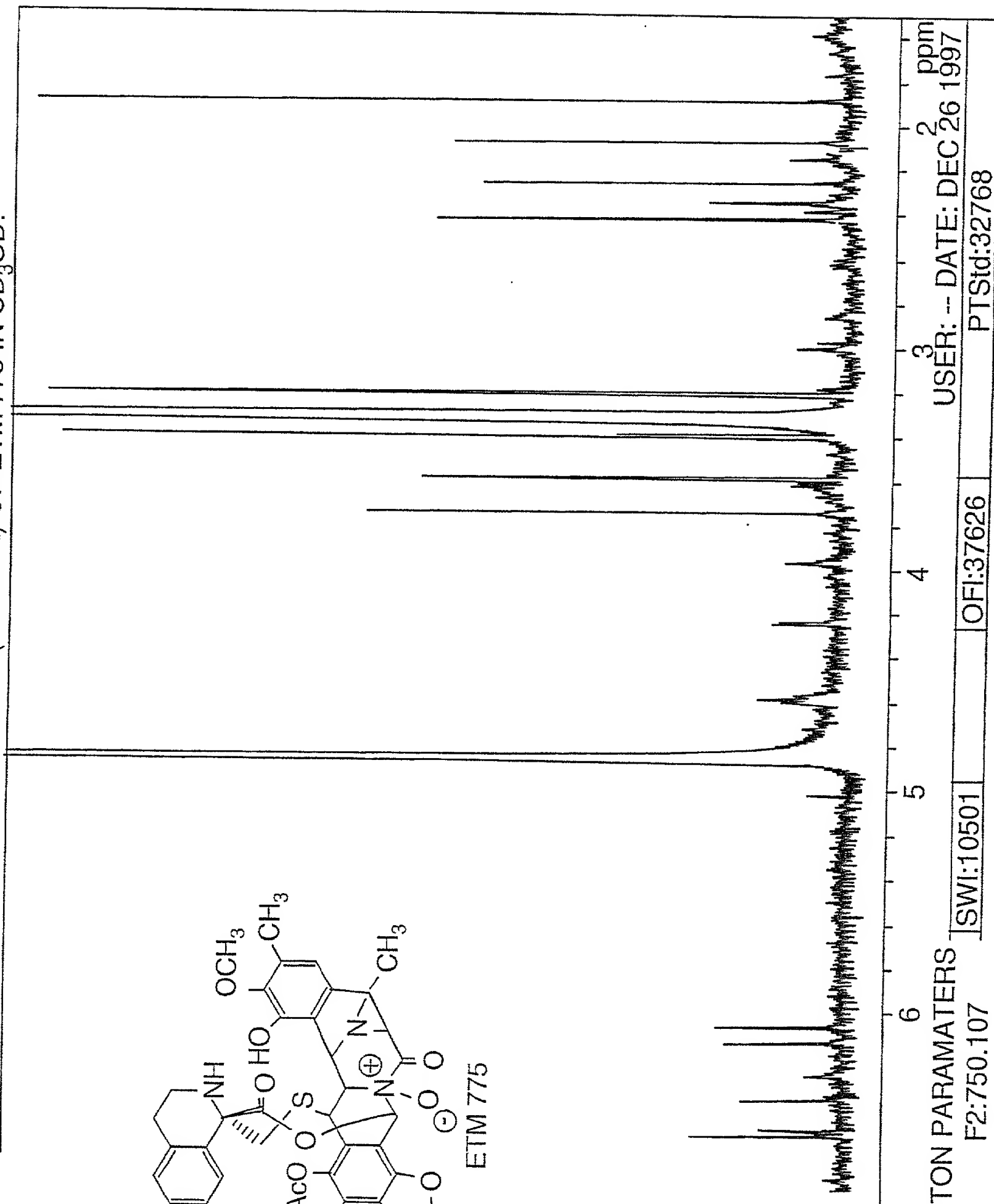


FIG. 16



DARD PROTON PARAMETERS

0.107

F2:750.107

SWI:10501

OFl:37626

6

5

4

3

2

ppm

USER: -- DATE: DEC 26 1997

PTStd:32768

UV SPECTRUM OF ETM 775.

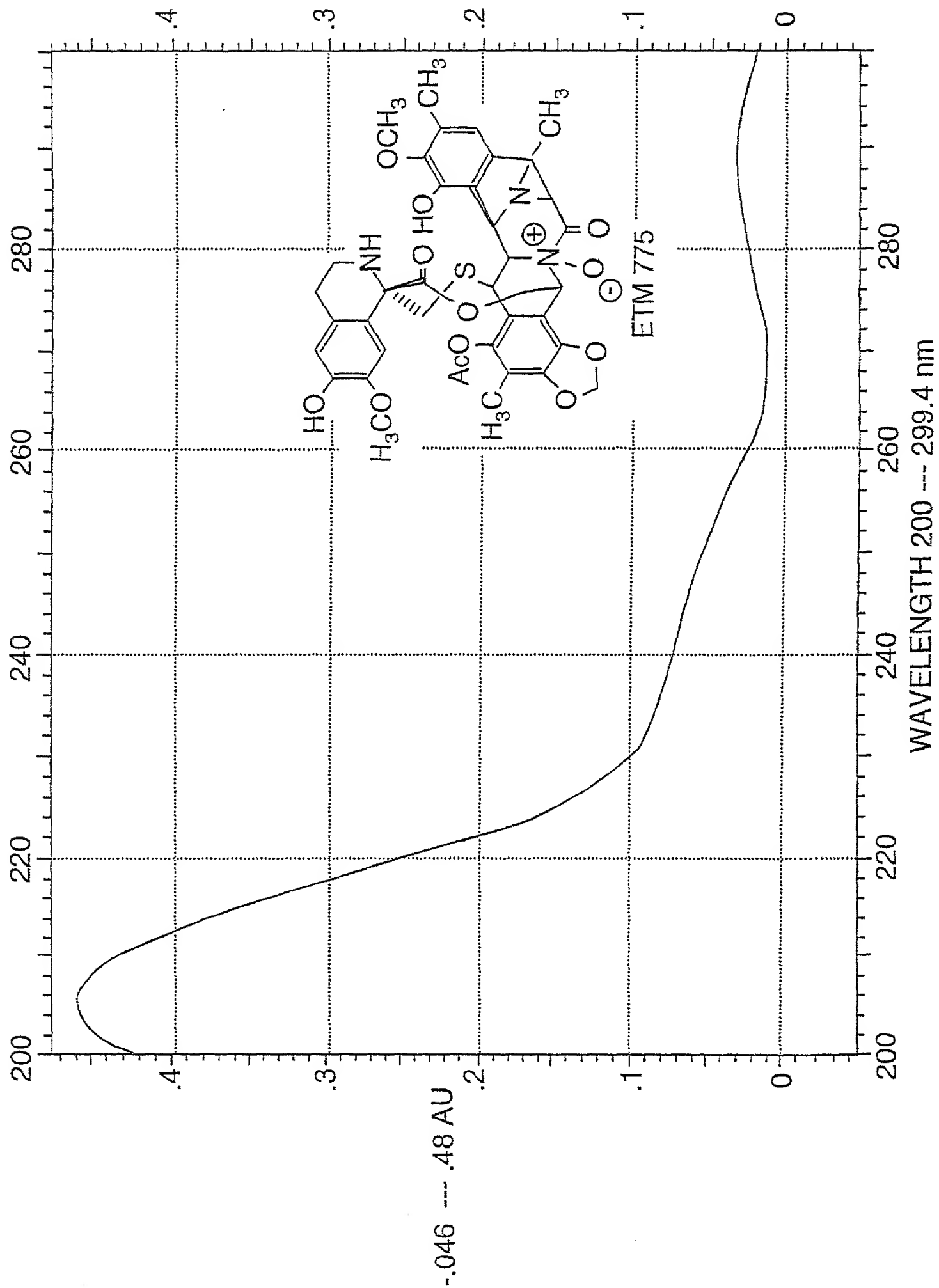


FIG. 17

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HPLC CHROMATOGRAM OF M1 METABOLITE (ETM 305).

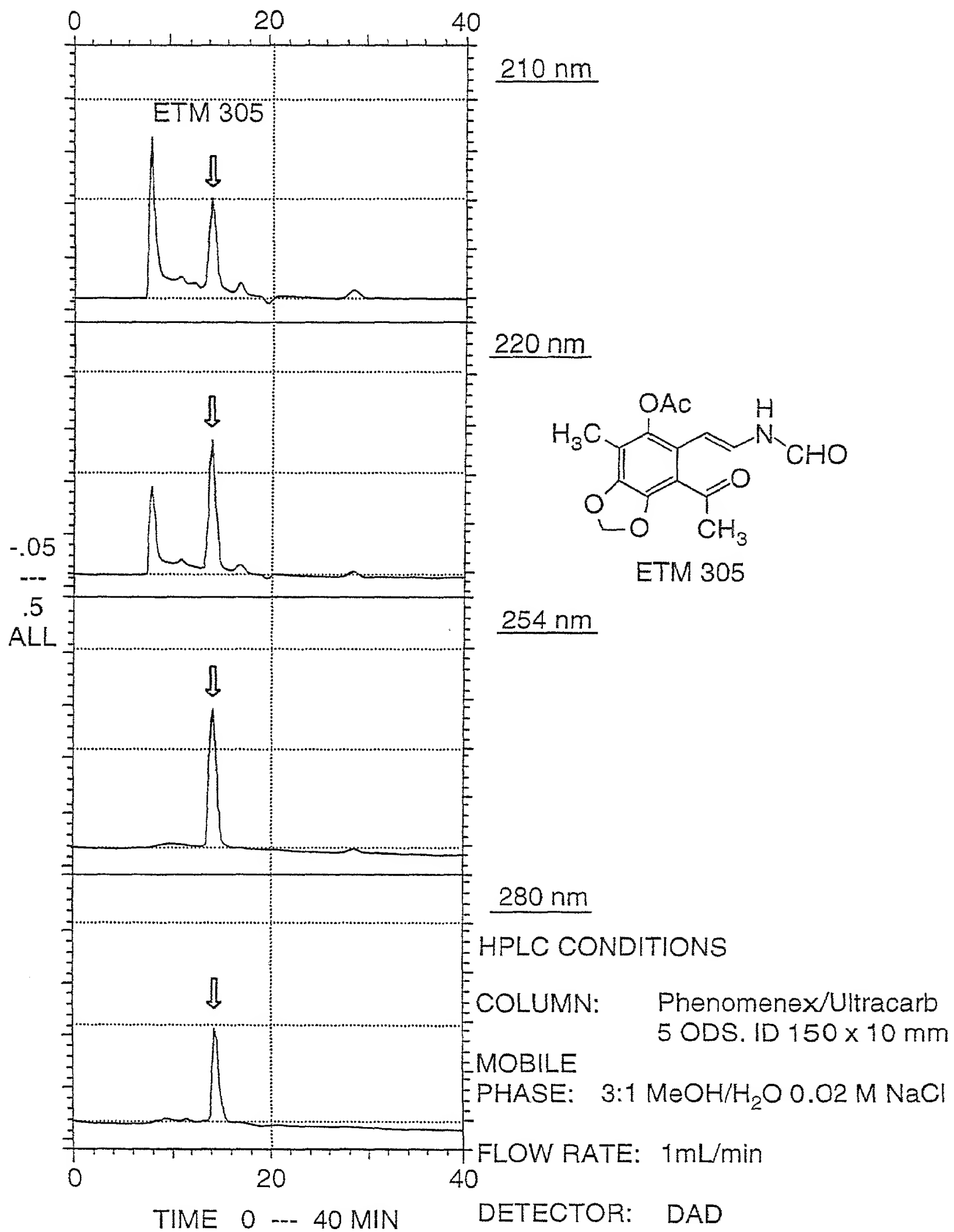


FIG. 18

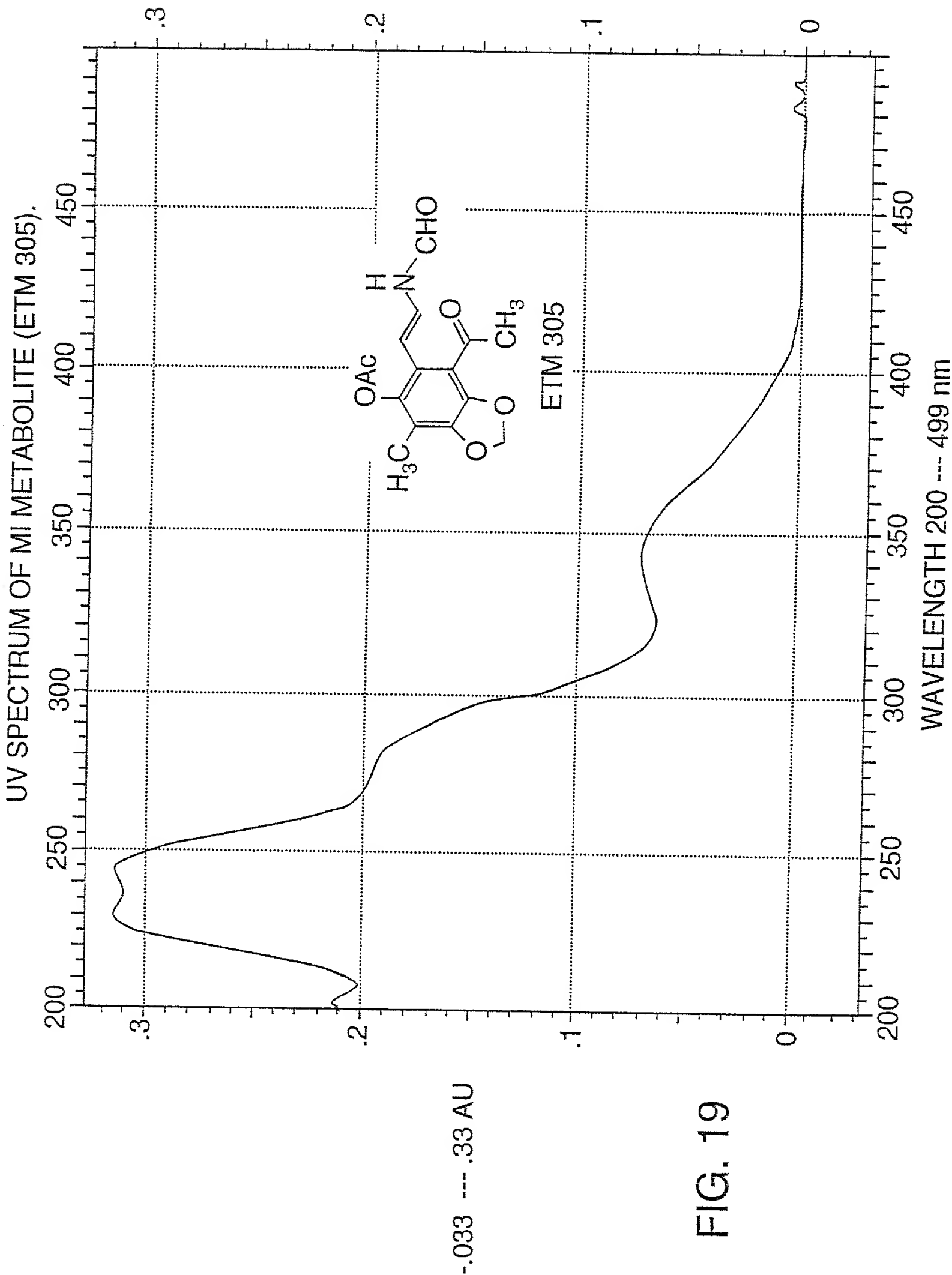
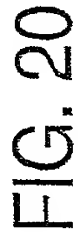


FIG. 19



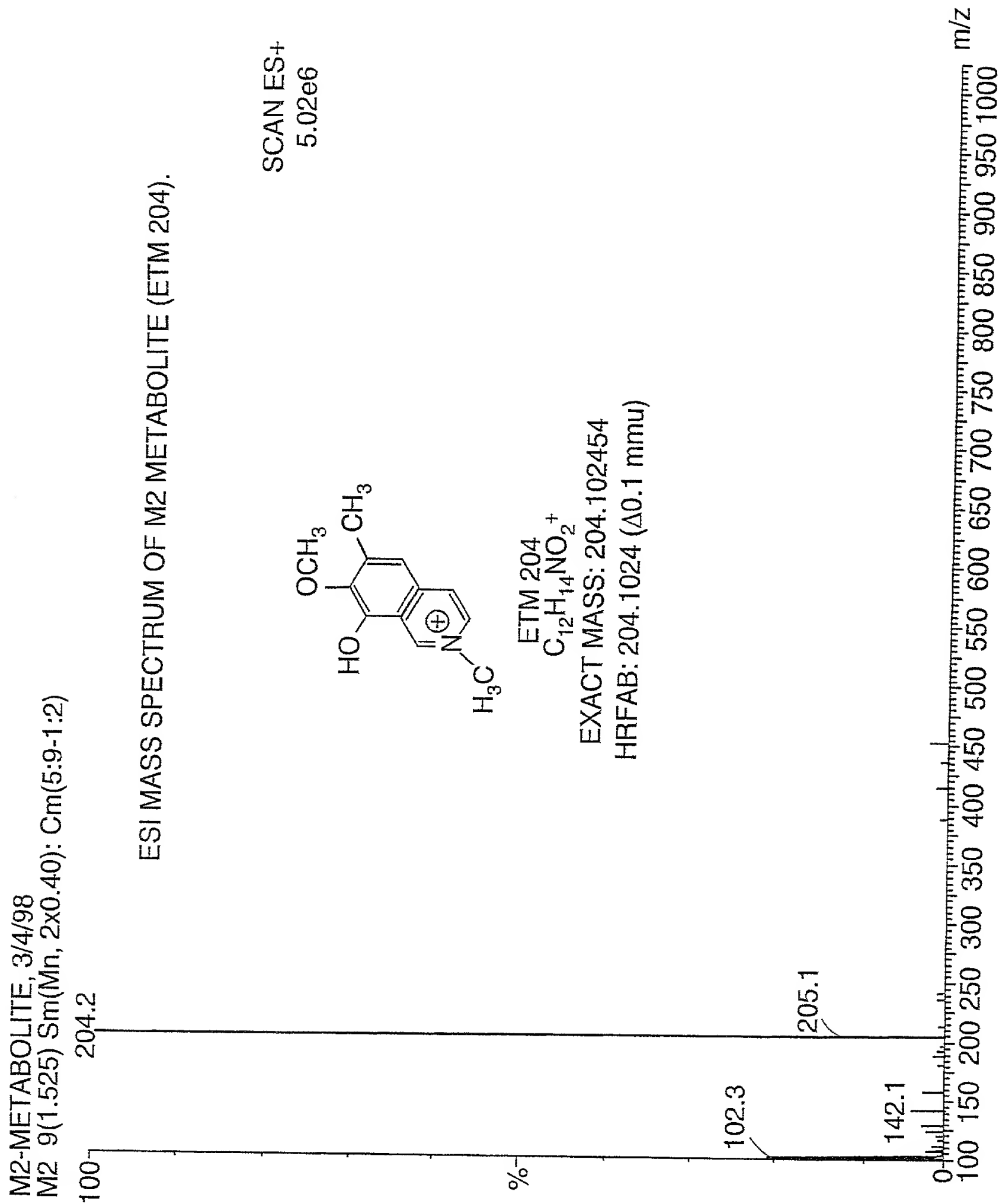


FIG. 21

FIG.
22AFIG.
22B

FIG. 22

MORALES, KLR, MZ IN CD3OD

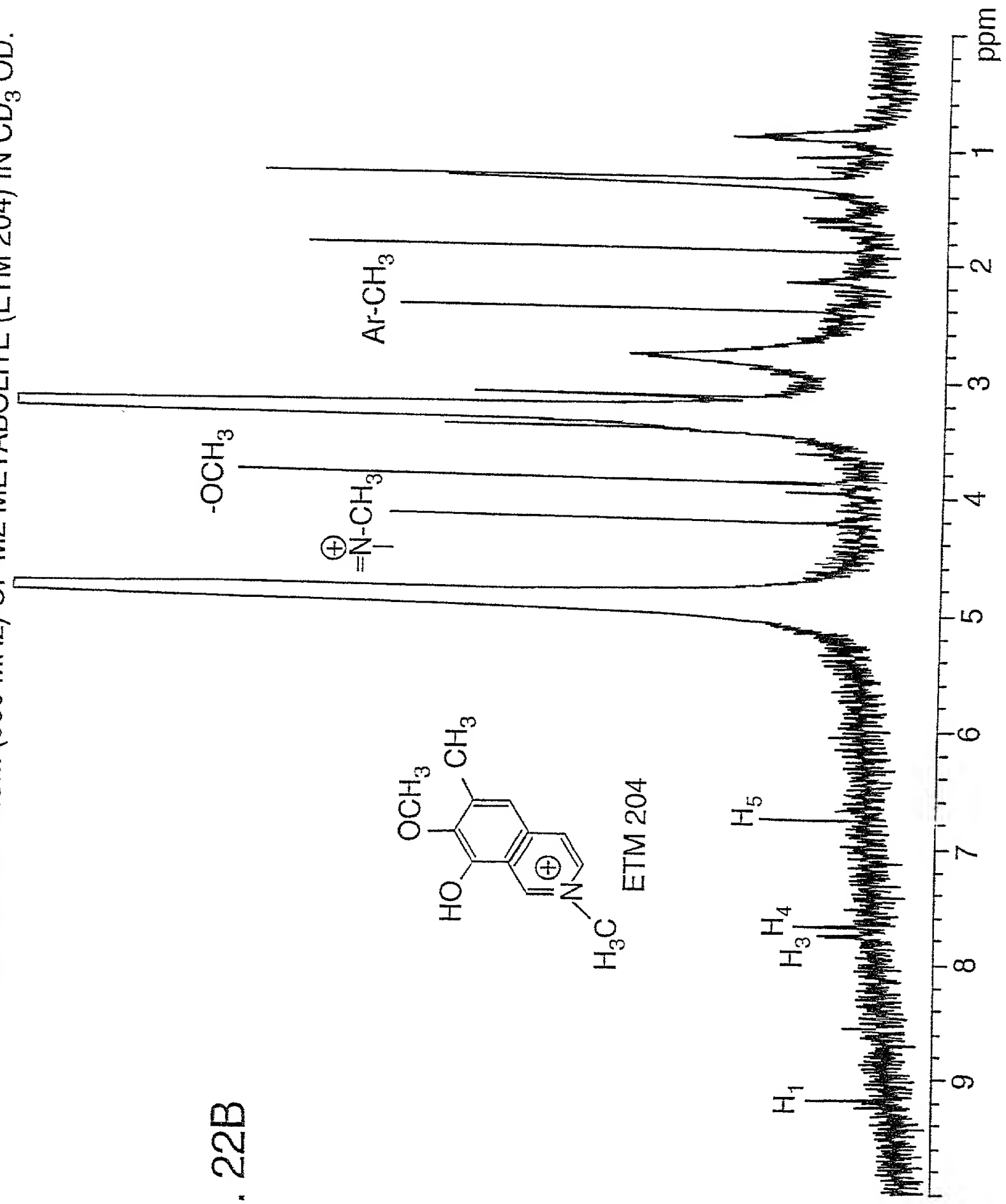
EXPL S2PUL

SAMPLE		DEC. & VT	
DATE	MAR 17 98	DFRQ	499.701
SOLVENT	METHANOL	DN	H1
FILE	EXP	DPWR	20
ACQUISITION		DOF	0
STFRQ	499.701	DM	NNN
TN	111	DMM	C
AT	4.003	DMF	200
NP	48000	DSEQ	
SW	5996.1	DRES	1.0
FB	3400	HOMO	N
BS	16	DEC2	
TPWR	63	DFRQ2	0
PW	4.5	DN2	
DL	0	DPWR2	1
TOF	0	DOF2	0
NT	3000	DM2	N
CT	1044	DMM2	C
ALOCK	N	DMF2	200
GAIN	NOT USED	DSEQ2	
FLAGS		DRES2	1.0
11	N	HOMO2	N
LN	N	PROCESSING	
DP	Y	LB	0.30
HS	NN	WTFIL	
DISPLAY		PROC	FT
SP	- 0.1	FN	NOT USED
WP	4997.0	MATH	F
V\$	31752		
SC	0	WERR	
WC	250	WEXP	
HZMM	19.99	WBS	
LS	33.57	WNT	
RFL	2154.5		
RFP	1649.0		
TH	7		
INS	1.000		
NM	PH		

FIG. 22A

¹H NMR SPECTRUM (500 MHz) OF M2 METABOLITE (ETM 204) IN CD₃ OD.

FIG. 22B



ESI/MS/MS SPECTRUM OF M2 METABOLITE (ETM 204).

M1, DAUGHT OF 204.2

M2D 1(0.109) Sm(Mn, 2x1.00): Cm(1:12)

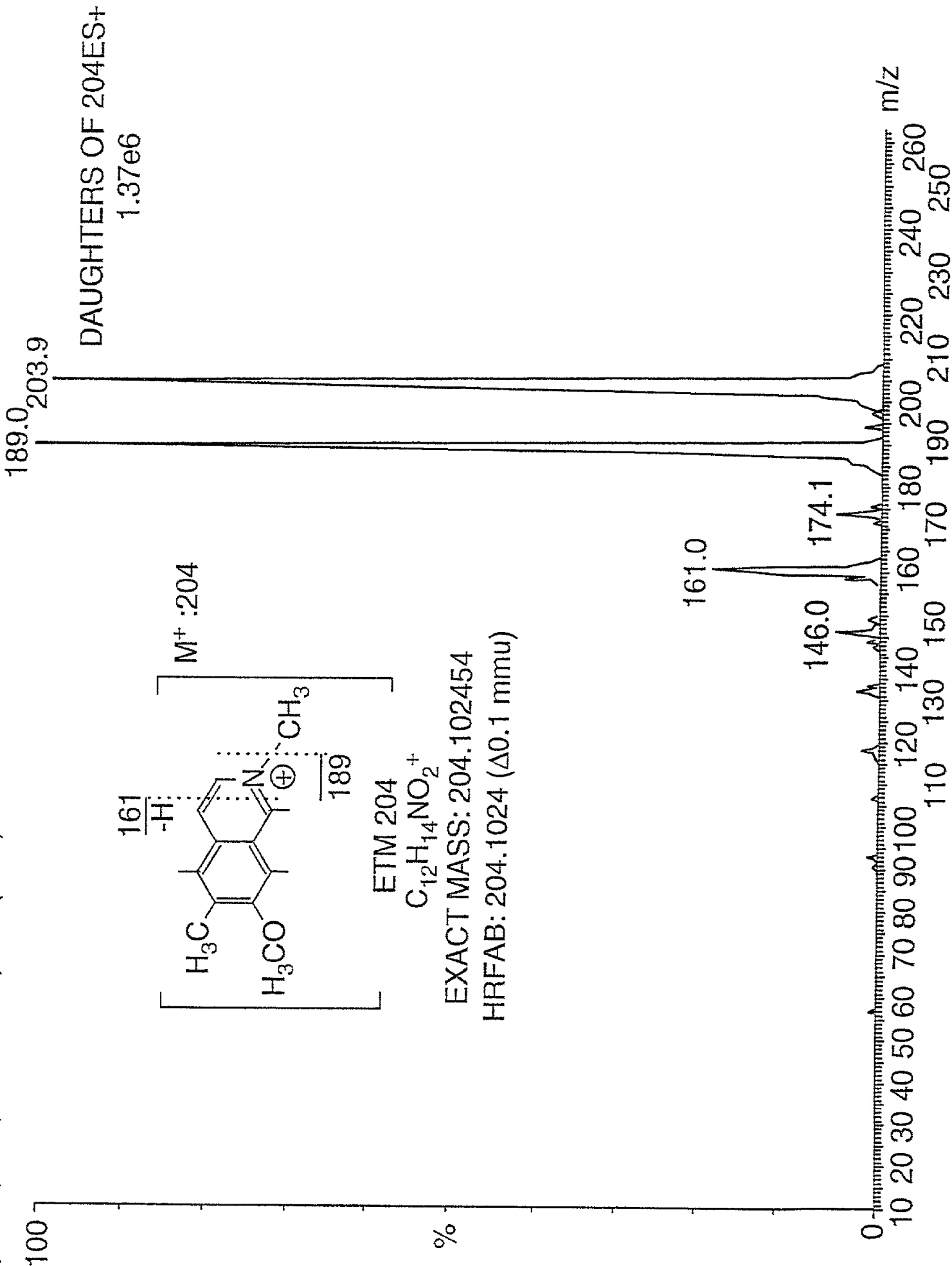


FIG. 23